# Luminescence dating inter-comparison for sediments associated with the Puente Hills Blind-Thrust System recovered from cores

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We present initial results gathered at the UCLA Luminescence Laboratory from OSL (optically stimulated luminescence) and IRSL (infra-red stimulated luminescence) measurements of samples collected from the Los Angeles segment of the Puente Hills Blind-Thrust Fault System, Los Angeles, CA, and compare them to OSL and C-14 measurements determined in other laboratories. In the University of Cincinnati Luminescence Laboratory, quartz OSL age estimates were determined for nine samples spanning a depth range from 3.5m-135.3m. At the UCLA Luminescence Laboratory, K-feldspar IRSL and quartz OSL measurements were made for five samples spanning a depth range from 9.3 m-135.3 m.

For the upper samples, the Cincinnati quartz OSL ages provide good agreement with the C-14 ages, while the UCLA K-feldspar IRSL age estimate for one sample may be too old. At greater depths, UCLA K-feldspar age estimates provide results that are in line with prior expectations based on extrapolation of sedimentation rate. However, the Cincinnati quartz OSL ages reach a maximum value of 35 ka, providing lower age estimates than expectation or K-feldspar IRSL. Assessment of the origin of the OSL signals from the quartz grains prepared at UCLA demonstrate that the signals are dominated by contributions from quartz, with no apparent inclusion of other minerals, such as feldspars or zircons. Therefore, the age variations seen in the quartz OSL ages compared to the IRSL feldspar ages may be due to sample contamination during collection, the use of different aliquot sizes or grain sizes in each laboratory, or other factors. Further experiments will be performed in order to determine what may be the cause of the apparent quartz OSL age underestimates, and to develop a reliable chronology for the Los Angeles segment of the Puente Hills Blind-Thrust Fault System, which will help us assess the potential earthquake hazard of the segment on metropolitan Los Angeles.

# EVALUATING A SAR TT-OSL PROTOCOL FOR DATING FINE-GRAINED QUARTZ WITHIN LATE PLEISTOCENE LOESS DEPOSITS IN THE MISSOURI AND MISSISSIPPI RIVER VALLEYS, UNITED STATES

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The Mississippi and Missouri river valleys in the midcontinental United States contain extensive loess-paleosol sequences that are used to constrain the timing of expansion and retreat of the Laurentide Ice Sheet. Previous studies have been unsuccessful in producing finite ages for sediments older than ~150 ka due to saturation of luminescence emissions. The thermally transferred optically stimulated luminescence (TT-OSL) dating technique is tested on the fine-grained (4-11 µm) quartz fraction of these loess deposits. The TT-OSL signal is found to increase with radiation dose up to 917 Gy. Optical sensitivity is measured with a sunlamp and bleach times are estimated at hours-to-days for total TT-OSL signal resetting. Equivalent dose values are highly sensitive to preheat temperatures, especially the 'thermal transfer' heating. Recycling ratios, zero-dose response values, and dose recovery tests all yield acceptable values for samples with IRSL equivalent doses > ~200 Gy (Forman and Pierson, 2002). The apparent TT-OSL ages for the Roxana Silt (~52-63 ka), Teneriffe Silt (~66 ka), and Loveland Silt (~133-192 ka) agree at 10 level with previously published TL and IRSL ages derived from the same samples(Forman and Pierson, 2002; Forman et al., 1992). For the oldest unit, the Crowley's Ridge Silt, TT-OSL ages (~167-200 ka) are younger than IRSL or TL ages This is interpreted as underestimation related to TT-OSL signal contamination, which can be avoided by isolating the fast component of the TT-OSL. Preliminary fast component TT-OSL ages for the Crowley's Ridge Silt (~197-241 ka) favor deposition during Marine Isotope Stage (MIS) 7 or 8, contrary to a previous inference of a MIS 12 deposition.

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# OSL dating of soils in University Valley, Antarctica: The dose-rate challenge

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The glacial history of small, hanging glaciers of the Quartermain Mountains, in the McMurdo Dry Valleys (MDV) of Antarctica, remains largely unexplored, even though most of these valleys contain geomorphologic features that can be used as paleoclimate proxies, including ground ice, rock glaciers, and patterned ground. We estimated the absolute age of soil samples from University Valley, a small hanging glacier adjacent to Beacon Valley, using OSL.

Two cores (Core 5 and 7) of ice-cemented soils were obtained ~20 cm apart at a location with shallow ice-table at 1 cm using a SIPRE corer, a hand-held coring system developed for coring into permafrost. The top 1 cm of overburden dry soil was collected to determine the resetting of the OSL signal. 5 samples in 20 cm intervals from Core 7 were used for OSL dating. Ice content was determined with high resolution from both cores. The ice content varied considerably throughout Core 7. There are also significant differences between Cores 5 and 7 although the cores were obtained within a short distance of each other. Nuclide concentrations varied as well, although no disequilibria in the Uranium decay chain were observed.

We will discuss our approach to calculating the effective dose rate. Measured water contents and nuclide concentrations from both cores were weighed by their distance from the respective OSL sample. Weighting factor was an exponential decay function and weights decreased with distance.

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#### INVITED ORAL PRESENTATION

# A NEW MEASUREMENT SYSTEM FOR LUMINESCENCE DATING - LEXSYG

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A highly flexible modular system for a variety of luminescence measurements used in dating application and luminescence research was developed. The LEXSYG systems provide standard measurements like Thermoluminescence (TL), Optically Stimulated Luminescence (OSL) and Infrared Stimulated Luminescence (IRSL), but also Infrared Radiofluorescence (IR-RF). A basic measurement chamber can be equipped with a variety of modules for irradiation, luminescence stimulation and detection.

The measurement chamber has a capacity of 80 cups/discs and is designed for vacuum/inert gas use. Samples are stored independent from the measurement section and hence sample change is possible at (nearly) any time, while radiation cross-talk, a common problem affecting the accuracy of luminescence dating, is avoided. The Beta (Sr-90) irradiation unit provides a homogeneous irradiation field with deviation across the sample area (10 mm diameter) of  $< \pm 3$  (about ±2% at 8mm) and allows RF imaging (8mm). Sample heating (TL/preheat) can performed up to 700°C. There several possibilities of OSL excitation by lasers and LEDs (850nm / IRSL; 532nm & 475nm / green & blue OSL; UV) which homogeneously illuminate the sample area for the stimulation of quartz and feldspar. The lasers can be used independently/simultaneously in CW modulated mode. The instrument configuration presented has 2 PMT (one standard bi-alkaline and an IR-sensitive cooled tube) and a cooled EMCCD based detection units. While the PMTs are used for conventional (small) aliquot measurements, the latter can carry out spatially resolved (resolution c. 16 µm/Pixel) / single-grain measurements of hundreds of grains at once. Two automatic optical filter changing units adapt relevant PMT or imaging optics (EMCCD) at the same time. Luminescence detection windows can be set from UV to NIR and new optical filtercombinations have been tested for particular emission wave-bands and luminescence types of feldspars and quartz to perform high efficiency luminescence measurements for experimental work and routine dating applications. Furthermore all detectors can be changed automatically between the TL/OSL and RF position also within a measurement sequence, allowing a maximum flexibility and high throughput. A solar light simulation unit allows mimicking of different natural bleaching modes with variable wavelength spectra of samples within the chamber.



Figure 1: lexsyg - luminescence measurement system.

# Interpreting D<sub>e</sub> distributions of single-grain data

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 $D_{\rm e}$  distributions from single-grain measurements are often characterized by over-dispersion which is higher than would be expected from a single-aged sample. How to interpret these distributions is not well understood. I present three case studies as illustrations.

- 1) Miracema do Tocantins is a paleoindian archaeological site within a sand dune in central Brazil (Bueno et al. 2012). Lithic vertical distributions show two Technology of the lithics suggests the two peaks represent discrete occupations; occurrence outside the peak regions is due to post-depositional movement, mainly upward. De distributions from five OSL samples also indicate post-depositional movement. Presumably sand grains have moved upwards as well. but the mechanism is unclear. If movement is from wind reworking, then solar bleaching is expected, and therefore the OSL, at least at minimum, should address age of reworking, not age of original deposition. Finite mixture modeling shows that the structure of the D<sub>e</sub> distribution varies considerably from sample to sample, suggesting very localized mixing mechanisms, perhaps from bioturbation. If grains are being moved up without solar bleaching, then the central tendency of the distribution should represent an over-estimation of the depositional age. If both wind reworking and bioturbation are at work, then the true depositional age should be bracketed between ages computed from the minimum age and central age models. These ages indeed bracket radiocarbon ages from charcoal collected in the peak locales. This suggests that OSL can confirm the radiocarbon ages but cannot provide precise age estimations by itself.
- 2) Sumiduoro is another paleoindian archaeological site in central Brazil, but located in colluvium on the shore of a lake (Araujo et al. 2012). Artifacts also are distributed vertically in peaks, but stratigraphic discordant radiocarbon dates suggest some mixing. Twelve OSL samples show high over-dispersion. Sediment analysis suggests that mixing is minor and the radiocarbon dates as a whole appear to be in stratigraphic order. In one section of the site, where there are five OSL samples collected from a column, the largest component of a finite mixture analysis gives ages in the right stratigraphic order and consistent with the radiocarbon chronology. OSL samples from other parts of the site are more badly mixed and can only give poor precision. Both OSL and C-14 are needed at this site for reliable dating.
- 3) Thirteen OSL samples, showing high over-dispersion, were collected from sandy archaeological deposits around Carolina bays in South Carolina. Both central and minimum age models produced ages in the correct stratigraphic order, but not the most common component of the finite mixture model. Independent evidence from radiocarbon and artifact typology agrees best with the minimum age results. There is no supporting evidence for partial bleaching, so some mechanism is moving old grains up a pattern that has been noticed by others in sand depositions and perhaps is not unusual for slowly accreting sand deposits.
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# Setting up luminescence dating protocols for sediments from the Negro, Solimões and Amazonas Rivers

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The Amazonas River and its tributaries form the largest sediment catchment basin around the world. Large amounts of sediments derived from denudation of the South American platform are temporary stored on rivers bars and floodplains before their transport to the Atlantic Ocean through the Amazonas River. In this context, dating the sediments stored within the Amazonas River System is important to understand the river response to climate, tectonic and relative sea level changes during the Quaternary. In this work, we present the procedures for Optically Stimulated Luminescence (OSL) dating of quartz regarding sample preparation and radiation dose estimation of sands from the Negro, Solimões and Amazonas rivers. The sediments from these rivers stand out due to their different composition and luminescence characteristics.

Samples were collected in trenches and outcrops of floodplains and bars. The sample preparation was performed under red light and consisted of: separation of 180–250 µm grain size through wet sieving; treatment with H<sub>2</sub>O<sub>2</sub> 29%, HCl 3.75%, HF 40% for 40 minutes, in order to remove organic carbon, CaCO<sub>3</sub> and feldspars, respectively; density separation with lithium polytungstate solution at densities of 2.75 g/cm<sup>3</sup> and 2.62 g/cm<sup>3</sup>. Initial measurements of the Negro River samples showed insignificant IR signal, indicating little feldspar content. However, the Solimões and Amazonas rivers samples showed high IR signals, demonstrating elevated feldspar content and their unsuitability for quartz separation using standard procedures. The observation of the Solimões and Amazonas sands under stereoscopic microscope showed 30% of lithic polimineralic sand grains composed of fine crystals of feldspar, mica and quartz. Aiming to eliminate the grains containing feldspar crystals, samples were etched twice with HF 40% for 40 min and 1h20min followed by re-sieving to discard grains below 125 µm grain size that were severely damaged by the HF treatment. Afterwards, IR signals decreased but still presented significant values. Then, we used the modified single-aliquot regenerative-dose (SAR) protocol by Wallinga et al. (2002) to measure the OSL signal of quartz in the presence of feldspar. The aliquots were stimulated with IR light at 175°C for 100s to eliminate the feldspar post-IR blue fast component and minimize the medium and slow components. The dose recovery test using this procedure after a laboratory dose of 8.80 Gy showed weighted average doses of 10.00±1.53 Gy for the Solimões sands and 9.29±1.2Gy for the Amazonas sands (12 aliquots per sample). This indicates that the quartz grains from the Solimões and Amazonas rivers have a good performance as radiation dosimeters even in the presence of feldspar contamination.

Despite their well behavior for the recovery of natural radiation doses, the doses estimated for quartz aliquots from the Solimões showed multiple modes with far more dispersion than would be expected from well-bleached samples. However, the doses estimated for the Negro River sands present low dispersion with single modes, representing well-bleached samples. This indicates that doses distributions depend on river dynamics and that a general classification of dating protocols for "fluvial sediments" should be avoided.

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Luminescence Characteristics and Experimental Methodology of Sediments from the Mecca Hills, California

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The Coachella Valley segment of the San Andreas fault in southern California has a "sawtooth" geometry in map view that consists of plate motion-parallel regions separated by regions of transpressive character. One of these transpressive regions, the Mecca Hills, has undergone significant deformation resulting in apparently tectonically offset geomorphic features, uplift related sedimentation, and variable geomorphic response to Neogene tectonics. Because the Mecca Hills exhibits strong potential as a neotectonic and paleoseismologic study area, there is a need to develop a protocol for dating tectonic sediments along and around the region. Optically Stimulated Luminescence (OSL) presents the most convenient option for dating geomorphic features, yet is complicated by several factors involving the luminescence characteristics of the sediment. Quartz grains recovered via standard sample preparation techniques seem to be "coated" with plagioclase feldspar and/or may contain significant plagioclase inclusions. Aliquots run using the standard SAR protocol often show a strong slow component in the quartz that may either by caused by instrisic mineralogical characterisitcs or plagioclase inclusions (IR prescreens suggest feldspar contamination is problematic in some samples). These findings are consistent with a source geology which includes highly arkosic sandstones and concentrations of minerals (i.e. monazites or apatites high in Th) that may create beta dosimetry "hot spots". However, infrequent aliquots show excellent decay curves dominated by the fast component, suggesting that at the quartz present within the sediment contains luminescence characteristics suitable for producing reliable dates if only it can be adequately separated from those inclusions and shown to have dosimetry that did not create luminescence inequalities. Efforts to extract a usable signal from the sediment around the Mecca Hills are ongoing and require further experimental research.

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OSL Dating of Ice-Marginal Sediments along the Green Bay Lobe, Wisconsin, USA

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To date there are very few reliable numerical age estimates that constrain the timing of the maximum extent of the Laurentide Ice Sheet in the upper Midwestern United States, a problem that stems in large part from the scarcity of datable carbon along the glacial margin. This study shows the results of dating glaciolacustrine and glaciofluvial sediments with OSL to determine when the Green Bay Lobe reached its maximum extent in south-central Wisconsin. Our study is unique in that geomorphic evidence suggests that each of our sites would only have been receiving glacial sediment during or shortly after the glacier stood at its maximum position. OSL work was done with the traditional SAR method using 90-150 um quartz grains mounted on 2 mm aliquots. We dated glaciolacustrine sediment found in lakes that were formed high in the Baraboo Hills where drainages were blocked when the Green Bay Lobe was at or near its maximum position. Unfortunately, our OSL results from these sites show a high degree of both inter-aliquot variability and spread in the final age estimates, but our OSL ages are in general agreement suggesting that the Green Bay Lobe reached its maximum position at  $\sim 18.5$  ka. At two other sites to the south of the Baraboo Hills we used OSL to date outwash that would have been deposited only when the Green Bay Lobe would have reached its maximum position. Our OSL ages show that outwash was deposited around ~ 23.4 to 21.4 ka. The earliest <sup>14</sup>C ages taken from glaciolacustrine sediments overlying these outwash deposits range between ~ 20.1-17.2 ka, supporting our OSL results at this site. Interestingly, all of our study sites are located within  $\sim 15$  km of the Green Bay Lobe, but show little evidence for partial bleaching. We expect to conduct additional work in the area to better constrain these events and further study the nature of bleaching at these ice-marginal sites.

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# Dating Exposed Surfaces using Penetration of OSL Bleaching

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Reduction of latent luminescence by exposure to sunlight, optical bleaching, is the basis for sediment dating. A popular new research topic is applying OSL to the surfaces of rocks or bricks to date when they were last exposed to light (Habermann et al. 2000, Greilich et al. 2002, Vafiadou et al. 2007, Viellevigne et al. 2006). One component of these studies is to look at the bleaching characteristics of the rocks (Habermann et al. 2000) to see how far beneath the surface light is able to bleach grains. This work explores whether the depth of bleaching into a translucent silicate rock can be used to determine how long a rock has been exposed to sunlight.

The analysis in this project utilized a model for OSL emission as a function of depth "x" given by:

$$I_{x} = \sum I_{0} \exp(-\sigma_{n} t \Phi_{0} \exp(-\alpha x)), \tag{1}$$

Where " $I_x$ " is the measured OSL emission at a depth "x" in the sample which is composed of the sum of one or more exponential components; " $I_0$ " is the OSL emission from the saturated unbleached material; " $\sigma_n$ " is the cross-section of the nth OSL component, "t" is time the surface of the material was exposed to sunlight, " $\Phi_0$ " is the in-band irradiance of the sunlight on the surface of the material, and " $\alpha$ " is the integrated absorption coefficient across the bleaching wavelength region.

Fifteen cores of quartzite cut from a sample of material from an actual prehistoric mine in west central Colorado received a dose of 1949 Gy at the Pacific Northwest National Laboratory, Hanford, Washington. This insured that the traps were saturated. Four of those cores were exposed to natural sunlight for 401 days at ATSAA in Denver, Colorado and one was exposed to the same environmental conditions but not sunlight for the same period, to provide a control. Two of the exposed cores and the control were examined at Scottish Universities Environmental Research Centre (SUERC) in Glasgow, Scotland using an experimental instrument that allowed vertical slices of the cores to be scanned in two dimensions to obtain a two dimensional mapping of the luminescence . The remaining two cores exposed to sunlight were sliced into twelve and sixteen, 400 micron thick disks respectively, and the luminescence measured using a standard SAR protocol at the OSL Laboratory at University of Washington, Seattle, Wa.

The luminescence data measured as a function of depth into the cores were analyzed by first decomposing the luminescence curves into three exponentials. The cross-section for each trap was determined from the fits. Using attenuation of the material measured on slices from other cores of the same material by both SUERC and ATSAA, an estimate of illumination time was obtained. This estimate did not agree with the actual illumination time. An apparent attenuation coefficient which was much less than the measured attenuation was calculated using the actual illumination time. It is our hypothesis that the larger attenuation measured by both SUERC and ATSAA was the result of scatter by the grains of the material as well as absorption. In the actual illuminated sample nearly as much light is scattered back into the material as is scatter away so the profile of bleaching with depth is due primarily to the attenuation of the material. This has led to definition of a protocol for using this method that includes repeating our controlled experiment on a sample of the material of interest to obtain a more reliable estimate of the apparent attenuation. This protocol is now being applied to a "blind test" to determine the illumination time of a core using the attenuation coefficient obtained by our controlled tests.

Timing of the Emplacement of Ancient Coastal Deposits of Georgia as Determined by ESR Optical Dating: A Work in Progress

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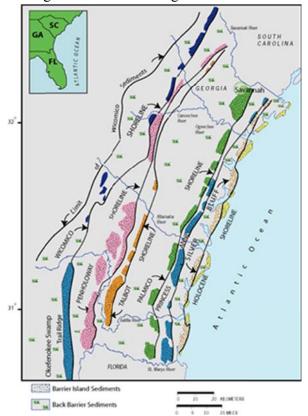
A number of coastal deposits have been mapped in Eastern Georgia and attempts have been made to determine the age of deposition of these deposits (Hails and Hoyt, 1969; Cornin et.al., 1984; Rhea, 1986). These studies have had limited success due to the information and technology that was available at the time. Using Electron Spin Resonance Optical Dating (ESR-OD) as well as Ground Penetrating Radar (GPR) a more detailed and complete picture of the events that occurred to emplace and preserve these deposits can be determined.

There are seven deposits that make up the ancient coastlines of Georgia. They are (from East to West or from low elevation to high elevation); the Silver Bluff, the Princess Anne, the Pamlico, the Talbot, the Penholoway, the Wicomico, and the Okefenokee (split in to the Okefenokee High and Low by elevation). The Wicomico and Okefenokees are also known collectively as the Trail Ridge Formation. While these names are used for deposits in Georgia and Southern South Carolina, deposits of different names from Southern Florida to north of Virginia are also sometimes correlated. Figure 1 shows the rough locations of these

deposits in Georgia.

The results of this study will contribute to the understanding of coastal processes in a number of ways. Determination of the age and position of these deposits will help to better understand the fluctuations in sea level. The age of these deposits will be used to define a system of correlation based on the timing of emplacement of each of the ancient coastal deposits. This will be used to relate the deposits found in Georgia the deposits found (and named differently) in Florida and further up the Atlantic Coast. Finally, determining the ages of these coastal deposits (when combined with the GPR survey) allows for hypothesis about how the river systems of Georgia developed. This will be a case study of how fluvial systems are affected by coastal processes.

Figure 1: The locations of the ancient coastal deposits of Georgia. Modified from Hails and Hoyt (1969).



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# Mitigating Problems Involved with OSL Dating of Arroyo Sediments in Kitchen Corral Wash, Southern Utah

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Kitchen Corral Wash (KCW), a tributary of the Paria River in southern Utah, has experienced both historic and pre-historic (Holocene) episodes of arroyo cutting and filling. During the most recent arroyo cutting event (~1880-1920), KCW and other regional drainages were entrenched up to 30m into their alluvial fill. This event left former floodplains perched above new channel bottoms and exposed stratigraphic evidence for past arroyo-cutting events along the channel walls. The sandy, quartz-rich sediments preserved within the exposed paleo-arroyos represent events of aggradation interrupted by periods of incision. AMS radiocarbon and Optically Stimulated Luminescence (OSL) dating will be used to better understand the timing of these events. While previous studies have attempted to constrain the timing of cutting and filling in KCW (Hereford, 2002 and Harvey, 2010), poor age control has limited results. The construction of a more detailed chronostratigraphy from KCW will help test hypotheses and determine if arroyo dynamics are synchronous between regional drainages, as proposed by Hereford (2002).

While research is still on-going, a total of fifteen OSL samples from eleven study sites will be analyzed for quartz single-grain dating using the Single Aliquot Regenerative (SAR) technique (Murray and Wintle, 2000). Recent studies applying OSL dating to arroyo-filled deposits (Bailey and Arnold, 2006; Summa, 2009; Harvey, 2010) identified partial bleaching as a significant problem. Partial bleaching was also expected here as KCW is an ephemeral drainage and is primarily influenced by floods from winter precipitation, spring meltwater runoff, and late summer flashy flows from monsoonal storms. The rapid, water-lain transportation of sediment in KCW over a short distance suggests that only a fraction of the grain population may have received adequate solar exposure to fully bleach (zero) the luminescence signal prior to deposition. In order to limit the affects of partial bleaching we followed the suggestions of Summa-Nelson and Rittenour (2012) and sampled fine-grained, well-sorted sand beds, <30cm thick, and/or containing ripple cross-bedding where possible.

Preliminary results indicate that singe-grain equivalent dose distributions have positive skew and high overdispersion, necessitating the use of the minimum age model (MAM) of Galbraith et al. (1999) to calculate preliminary age estimates. The stratigraphy of key arroyowall exposures containing multiple cut-fill sequences will be presented with new AMS radiocarbon ages and preliminary single-grain OSL results.

Experiences in assessing the abundance of K in feldspar

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In recent years we have seen a renewed interest by many laboratories in using K-feldspar minerals for luminescence dating. This will increase the amount of behavioral and physical investigations on this mineral on certain topics such as anomalous fading. One parameter specific to feldspar is often overlook, namely its abundance in K inside the crystalline matrix. The issue is relevant not just to K-feldspar but also plays an important factor for Na- and Ca-plagioclases. For K-feldspar, although unlikely, there is the risk for the sediment to lack microcline/orthoclase minerals and be made up of low K-feldspar, such as anorthoclase.

Its measurement is rarely performed since available techniques, such as beta counting or high-resolution gamma spectrometry, typically requires a gram of material. One problem that arises with these two approaches is that it is difficult to concentrate 1 gram of feldspar.

Here we will present some recent results we obtained in assessing the abundance of K in feldspar by using various techniques: high-resolution gamma spectrometry, XRF, X-ray diffraction, EDS (energy dispersion imaging) and electron microprobe.

Equivalent dose distributions demonstrate that varied fluvial transport processes are a major component in the construction of floodplains

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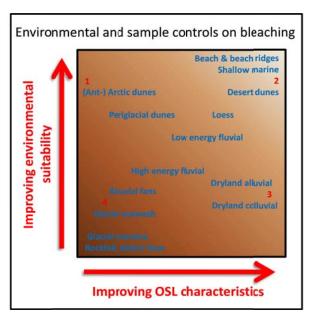
In the Buffalo National River, depositional ages of coarse-grained quartz sand sampled at a variety of locations and depths within one discontinuous floodplain were determined using OSL. The variation in the distributions of equivalent doses  $(D_e)$  for different sample locations yields insight to the process of floodplain formation. Where  $D_e$  distributions show high over-dispersion and the likelihood of partial bleaching, sediment is likely transported as bedload and deposited in point bars as the channel aggrades laterally. Where  $D_e$  distributions are normal, sediment is transported in suspension during high flow events or is deposited as overbank sand dunes on the floodplain, resulting in complete bleaching of the sediment package. The spatial distribution of ages suggests a long period of interaction between lateral channel migration and vertical aggradation of the floodplain through overbank flooding.

# TITLE

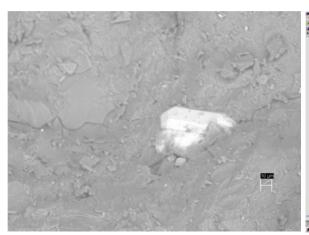
Lawson, Michael J., Rhodes, Ed J., Ochoa, Lupe, Barrera, Wendy A.

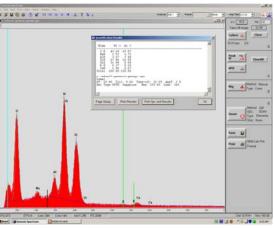
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Conventional quartz OSL (optically stimulated luminescence) can be hindered in tectonically active regions by a number of factors including low sensitivity, reduced bleachability and signal contamination. These problems can be compounded in high energy depositional contexts that affect signal resetting (Fig. 1). As contributions from very small volumes of



mineral inclusions (Figs. 2, 3) within quartz grains can significantly affect OSL behavior, and potentially lead to erroneous age estimation, we have developed a test designed to quantify the degree of signal contamination which will be presented. Four factors will be explored, including the thermal quenching factor, post IR OSL ratio, IRSL/ OSL<sub>IRSL</sub> ratio and IRSL intensity, which when used together serve as powerful diagnostic tests on the purity of a quartz sample.





# A well-preserved sequence of high-elevation ice age ecosystems: Can the Snowmastodon lacustrine sediment be dated using optically stimulated luminescence?

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In October 2010, a bulldozer operator working near a Colorado ski area uncovered the tusk of a young female mammoth. Over the next 10 months, the Denver Museum of Nature & Science conducted its largest-ever fossil excavation at the site that came to be known as Ziegler Reservoir, yielding a treasure trove of well-preserved Ice Age fossils. Museum crews uncovered 5,000 bones of 41 kinds of Ice Age animals, including mammoths, mastodons, ground sloths, camels, deer, horses, and giant bison. The preserved series of Ice Age fossil ecosystems is one of the most significant fossil discoveries ever made in Colorado.

To better understand paleoclimatic conditions and to provide a chronological framework for the Snowmastodon, CO paleontological site, we undertook a study of the glacial basin stratigraphy using Optically Stimulated Luminescence (OSL) dating. OSL dating provides a method of determining the burial conditions and age of sand grains entombed in the lake sediments. Eleven samples were collected at various depths and locations throughout Ziegler Reservoir during the June 2011 excavation, and later, an additional six samples were collected from sediment encased in mammoth tusks or from large intact blocks of sediment that were collected for pollen analyses. The fine-grained quartz sand (90 to 63 microns) was extracted from the samples under dark room conditions and analyzed using single aliquot regeneration techniques (SAR). Environmental dose rates were estimated and compared using elemental concentrations from ICP-MS, gamma spectrometry, neutron activation, and on-site gamma spectrometry.

Samples were taken in a vertical profile from several sites and in a cross-section of the lake for Unit 10. OSL ages range from  $69.5 \pm 4.2$  ka for the uppermost layer sampled (Unit 17), to  $148 \pm 7.69$  ka for the lowermost unit sampled, (Unit 8). Unit 10, nicknamed as "the yellow brick road" was sampled from three locations and produced OSL ages of  $91.8 \pm 4.79$ ,  $100 \pm 6.72$ , and  $105 \pm 5.82$  ka for the middle, south, and north sections of the basin. These ages are stratigraphically consistent and support the hypothesis that the creation of the damning moraine is a result of Bull Lake age glaciation.

OSL characteristics for the quartz displayed excellent decay curves with little to no slow component and no evidence for thermal transfer, suggesting a source of mature sedimentary material, possibly eolian in origin. Although the OSL ages are old (close to 100 ka in many cases), there is no compromise in the signal due to saturation of the luminescence trap sites within the mineral grains. When the equivalent doses are plotted in statistical programs they show very little scatter, indicative of eolian origin for the sand-sized grains. Where scatter is apparent (Unit 17 and Unit 8), the simplest explanation seems to be that there may be more of an alluvial component than an eolian component to the sedimentation in the unit. Ages within the stratigraphy are often closely spaced and within error, suggesting that the sediment accumulated in the basin on timescales of 10's of ka thus contributing to the excellent preservation of the paleontology. Ages of sediment within the bone beds, thus dating the time of the majority of the fauna specimens, will be presented.

# IRSL Measurement Applications for Sediment Transport Processes: Mojave River Case Study

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The Mojave River system was chosen as a case study to develop applications of feldspar IRSL measurement techniques for sediment transport processes. A previous hydrologic study by Enzel & Wells (1997) shows that significant flow (defined as >90m<sup>3</sup>s<sup>-1</sup>) frequency decreases down the river. The frequency of significant flow events at the Afton Canyon gauging station, near the termination point of the river, is about once every ten years, closely correlated with El Nino events. In this study, the IRSL signals of K-feldspar grains (175-200 μm) were measured at temperature increments 50-95-140-185-230 °C over multiple cycles using a modification of the Post-IR IRSL procedure developed by Buylaert et al. (2009). An experiment to test for bleachability was carried out by placing subsamples in direct sunlight for increasing amounts of time. This experiment was used to determine bleachability ( $\alpha$ ) and kinetic order ( $\beta$ ) coefficients. Bleachability was observed to increase with the number of exposure events. IRSL measurements of 8 modern channel samples along the river were used to determine equivalent dose (D<sub>e</sub>). A general decreasing trend was observed from the headwaters of the Mojave River to Afton Canyon, near the terminus. The ratio of IR-230 °C De to the De of IR-50 °C decreases linearly with distance downriver. The decrease in ratio reflects the increasing bleaching of higher energy (deeper) electron traps with distance downriver, suggesting cyclical exposure of grains as they are transported. The geographic trend was compared with a simple numerical model, which allowed for discrete bleaching events over 10 year and 100 year time steps. The simultaneous fit of model iterations with the data points for IR-50, IR-230 and IR-230:IR-50 ratio supports the hypothesis that events of brief daylight exposure (<400 s) were followed by longer periods of complete burial (no exposure; IRSL signals growing). A rough estimate of a minimum time constraint on sediment transport yields a provisional transport rate of 100m downstream per year. Further research will include additional sampling and more sophisticated numerical and analytic solutions minimizing cumulative time to fit equivalent dose observations of the measured dataset.

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Using OSL and Radiocarbon Dating to Constrain the time of soil development MIAO Xiaodong<sup>1</sup>, WANG Hong<sup>1</sup>, HANSON, Paul R<sup>2</sup>, MASON, Joseph A<sup>3</sup>

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Time, one of the five factors in soil development along with climate, parent material, organisms, and topography, is theoretically defined as the time elapsed since the parent materials were deposited and subaerially exposed, according to Jenny's model. Soil time was estimated previously based on the degree of soil development, but in many environments the soil ages have not been practically calculated.

We propose that the best method for estimating the time of soil development is subtraction of the Pyrolysis-Volatile (Py-V) <sup>14</sup>C dates of soil's uppermost A horizon from OSL dates of C horizon of parent material. The Py-V <sup>14</sup>C dates represent most mobile soil organic carbon that is least resistant to biodegradation in soil environment, therefore yielding youngest ages potentially, while OSL dating on the C horizon estimates the depositional time of the parent material.

We tested this new approach in four scenarios. 1. Modern soil developed downward in loess. Results from this simplest case show that modern soil formed in Peoria loess in central Illinois (at the Mahomet site) was exposed at the surface for about 16 kyr. 2. Soil develops while loess aggrades. When rate of soil development exceeds that of dust deposition, a very thick cumulative A horizon forms and soil time should be the whole duration of the cumulative soil development. The OSL date of the uppermost C horizon is best used for this calculation. Results from Brady Soil in Nebraska of the central Great Plains (at the Wauneta site) show that the Brady soil developed for about 6 kyr. 3. Soil formed in dune sand. Unlike loess, eolian sand can deposit rapidly in one single event, and previous studies have revealed that a few meters of dune sand can be deposited within very short period in the geological time scale. Thus, age from the uppermost C horizon is not required. Instead, statistical average of the dune sand ages, including the ones well below the solum, provides a better estimation of the parent material age. In the dune sand of Green River Lowland of Illinois (at the Atkinson site), the average of three OSL ages from soil C horizons (17.7 kyr) minus the Py-V <sup>14</sup>C date of 2.7 kyr from upper most A horizon yields a soil time of about 15 kyr. 4. A counterexample shows that simple subtraction is not always appropriate. An organic rich gyttja deposit was dated in the Havana Lowland dune field in Illinois (at the Manito site). The Py-V date of 13.2 kyr overlies the loess that was dated with OSL at 14.1 kyr. Clearly, soil time is not 0.9 kyr because the two ages are not obtained from the same parent materials. Therefore, caution must be taken when applying this method.

This approach constrains soil time more reasonably than using either OSL or radiocarbon dates alone. If no old carbon contamination is assumed, the difference between the Py-V ages at the uppermost and lowermost sola of a soil almost certainly underestimates the time of soil development, and the difference between OSL ages taken from above and below a soil most likely overestimates it. Combination of OSL and radiocarbon can best constrain soil development time.

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# Non-Linear Light Modulated OSL Phenomenon and Thermally Assistance OSL Measurements

The new methods for improving dose estimation from α-Al<sub>2</sub>O<sub>3</sub>:C

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A new Non-linearly modulated (NL) Optically Stimulated Luminescence (OSL) measurements technique has been developed which gives greater signal to noise ratios apart from adding more OSL defects related information. A theoretical simulation and experimental verification of non-linear light modulation of optically stimulated luminescence (NL-OSL) technique has been demonstrated. The NL-OSL technique can be useful in analyzing OSL traps having very low and closely lying values of photo-ionization cross-sections of meta-stable traps obeying different order of kinetics. Recently, we have also given a detailed study of non-linear optically stimulated luminescence (NL-OSL) phenomenon on  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>: C. The experimental values of signal to noise ratio have been compared for

NL-OSL and LM-OSL modes of stimulation using  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>: C phosphor. The experimental results show definite advantage of NL-OSL over LM-OSL and CW-OSL, as NL-OSL technique has been found to be useful in separating fast OSL component more effectively, thus leading to extended dose linearity from the OSL defects having larger photo ionization cross-section, particularly in  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>: C, with added feature of multiple dose assessment for single irradiation.

In order to understand nature and physics of phonon coupling with OSL trap levels, the method of measuring thermal assistance energy associated with photo-ionization cross-section of

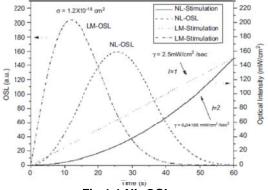


Fig.1.1 NL-OSL

 $\alpha$ -Al<sub>2</sub>O<sub>3</sub>:C has been suggested by us. This method involved recording of CW-OSL by stimulating simultaneously with 470 nm light and temperature using linear heating, the process termed as Thermally Assisted (TA)-OSL. The theoretical modelling has been done for the observed TA-OSL phenomenon in  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>:C phosphor. The TA-OSL signal for  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>:C has been investigated for various stimulation intensities and different heating rates. The validity of proposed theoretical relation on temperature dependent nature of photo-ionization cross-section has been investigated using experimentally determined values of thermal assistance energy ( $E_A$ ) and temperature independent pre-exponential factor of photo-ionization cross-section.

Under accidental / In light dosimetry the commercially available  $\alpha$ -Al $_2O_3$ :C powder was studied for deep energy level defects by a newly suggested method using thermally assisted optically stimulated luminescence (TA-OSL) phenomenon. The method involves simultaneous application of continuous wave optically stimulated luminescence (CW-OSL) as well as thermal stimulation up to 400 °C, using a linear heating rate of 4 K/s. By using this method, two well-defined peaks at 121 °C and 232 °C were observed. These TA-OSL peaks have been correlated to two different types of deeper defects which can be bleached at 650 °C and 900 °C respectively on thermal treatment. These deeper defects, having larger thermal trap depth and relatively lower photo ionization cross-section at room temperature for stimulation with blue LED (470 nm), are stable up to 500 °C, so they can store absorbed dose information even if the sample is inadvertently exposed to light or temperature. As only a fraction of signal is bleached during TA-OSL readout, multiple readouts could be performed on an exposed sample using this technique. The dose vs TA-OSL response from deep traps of  $\alpha$ -Al $_2$ O $_3$ :C was found to be linear up to 10 kGy.

<u>D.R. Mishra</u>, M.S. Kulkarni, N.S. Rawat, Anuj Soni, B.C. Bhatt, D.N. Sharma. 2011 Preliminary non-linear light modulation OSL studies using  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>: C *Radiation Measurements 46*, 1462-1468.

# Illustrating the versatility of portable OSL readers: case studies from the Canadian Prairies

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The use of portable optically stimulated luminescence (POSL) readers to elucidate on complex depositional sequences has been demonstrated in a number of recent studies (e.g. Sanderson and Murphy, 2010; Munyikwa et al., 2012). POSL readers are robust devices that can be used in the field, allowing for rapid decisions to be made when collecting samples for OSL dating. The readers can also perform measurements on bulk samples, negating the need for time-intensive mineralogical separations. POSL readers are equipped with both IR and blue light stimulating sources such that signal separation during measurement can be achieved by selectively exciting feldspar using the IR source after which a quartz dominant signal is obtained using post-IR blue OSL. Typically, the acquired signals are then plotted to give luminescence profiles that depict the variation of the luminescence signal with depth. When all the variables that control the luminescence signal up the depositional sequence are held constant, apart from the burial age, the luminescence profile serves as a proxy for the chronostratigraphy.

To illustrate the versatility of POSL readers, this presentation outlines three studies in which a POSL system developed by the Scottish Universities Environmental Research Centre has been used on postglacial and Holocene eolian dune terrain in Alberta, Canada to gain better insights into the stratigraphy. In the first study, we used the POSL reader to construct luminescence profiles of the dunes in order to delineate cryptostratigraphic interfaces that are otherwise difficult to identify (Munyikwa et al., 2012). Results show that luminescence profiling enables one to distinguish between eolian deposits that make up the dunes from the underlying glaciofluvial sands because the glaciofluvial deposits were not well bleached prior to burial.

In a separate investigation at an archeological site in southern Alberta, we used a POSL reader to create luminescence profiles of the shallow subsurface in an effort to demarcate areas of the site where the eolian stratigraphy has been disrupted by oilfield pipeline construction. Disturbed areas return luminescence profiles with signals that fluctuate with depth due to sediment mixing whereas the signal profile from areas with intact stratigraphy is more stable. In the third study, we used the POSL reader to approximate relative ages of depositional units in stratigraphic profiles. Overall, these studies demonstrate the unlimited potential of POSL readers.

#### References

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Munyikwa, K., Brown, S.T., Kitabwalla, Z. 2012. Delineating stratigraphic breaks at the bases of postglacial eolian dunes in central Alberta, Canada using a portable OSL reader. Earth Surface Processes and Landforms. DOI: 10.1002/esp.32

Defining the timing and rates of floodplain deposition controlled by climate and tectonics along the lower Ohio River

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The nature and timing of floodplain deposition was examined along a stretch of the lower Ohio River near Evansville, Indiana. The Uniontown fault is a recently discovered active fault that traverses the floodplain, and it was examined to assess its deformation history and control on floodplain deposition. The fault's long-term slip history was investigated by mapping, ground penetrating radar, electrical resistivity surveying, fault trenching, coring, and optically stimulated luminescence dating. Samples for OSL dating were collected across the fault and the floodplain using a trailer-mounted Giddings 25-SCT HDGSRPST hydraulic soil sampling coring rig. A transect of eight sediment cores between 9 and 13.5 m deep were obtained across the scarp to develop a stratigraphic framework of deeper sediments, and OSL samples were collected from the floodplain surrounding the scarp at more than 25 sites. For each sample 24 aliquots were measured and ages were determined using single aliquot regenerative method (SAR). All samples have high signal to noise ratio and results were obtained with a clustered histogram oriented to mean. OSL ages of deposits and landforms around the scarp cluster into four distinctive time intervals: late Pleistocene to early Holocene (15.8 –8.2 ka), early Holocene (10.1 – 5.4 ka), early to mid-Holocene (8.9 – 3.7 ka), and late Holocene (2.19 – 1.9 ka). The late Holocene deposits post-date faulting, indicating the timing of the most recent movement on the Uniontown fault was between 5.5 and 1.5 ka. which is also when the modern channel course of the Ohio River was established around the Uniontown fault. This suggests that activity on the Uniontown fault altered the course of the Ohio River. The OSL chronology identifies four phases of fluvial landform development since the late Pleistocene, and one or more of these could represent fluvial adjustments to older paleoearthquakes.

The pros and cons of single grain K-feldspar IRSL sediment dating in neotectonic and paleoclimate contexts

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In regions where quartz OSL characteristics are not well-suited for making optical dating measurements owing primarily low signal sensitivity, K-feldspar offers a promising alternative target mineral group. Expected issues are i) anomalous fading of luminescence signals, and ii) potentially lower daylight bleaching rate in comparison to quartz. Several sites in Southern California have been dated using the IRSL signals of sand-sized single grains of K-feldspar, based on a post IR IRSL protocol, similar to that of Buylaert et al. (2009). The protocol adopted uses a preheat following natural, regenerated and test doses of 60s at 250°C, 3s IRSL at 90% laser power for each grain at 50°C and subsequently at 225°C, a 3.7 Gy test dose, the same preheat and IRSL measurements to measure the test dose response, and a final hot bleach for 40s at 290°C using IR diodes in a Risø TL-DA-20-D reader. Besides equivalent dose determination for each grain, a further challenge is to determine anomalous fading properties in terms of g-values. Measurements of several samples reveal an interesting pattern of single grain De distribution in the form of a systematic increase in minimum De value with increasing grain sensitivity. A provisional interpretation of this pattern is that for sediment samples in contexts where post-depositional grain mixing is unlikely, the minimum values represent the best-bleached grains, and that lower dose values for dimmer grains represent the effects of an increased degree of fading. The implications of this interpretation for dating sediments are considered, and evidence for any systematic dependence of g-value on grain sensitivity examined.

Subterranean transport and deposition of quartz by ants in sandy sites relevant to age overestimation in optical luminescence dating

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An artificial layered sandy site was created using a combination of native sand and colored sand. Twelve layers of sand, each 1 by 1 m in horizontal extent by 10 cm in thickness were emplaced to a depth of 2 meters followed by implantation at the surface of a Florida harvester ant (Pogonomymrex badius) colony. The colony excavated a nest, and then after 7 months, the layers were excavated to test the hypothesis that sand grains were moved upward within the ant nest without reaching the surface. The ants penetrated 11 of the 12 colored layers reaching a depth of 130 cm. More than 16,000 colored grains were identified in layers that did not originally host them. Of these, more than 80% were unambiguously moved upward. This means that possibly as many as 54,000 upwardly mobile grains were present (ratio of 3:1 uncolored to colored). In relation to optical luminescence (OSL) dating, this means that grains that would not have been optically zeroed by transport to the surface (defined here as subterranean-transported) were present in abundance, and that if the site was ancient, there would have been found many grains that were older than the layers they presently reside in, even if only one colony of harvester ants had disturbed the layers. We conclude that ants can significantly affect the age distributions in sandy archaeological sites. Multiple examples of such disturbances

have been documented in the literature. Most relevant to our results are recent studies of the OSL chronology of Preand Palaeoindian Clovis-age archaeological sites in sandy environments in North America that may have been compromised by ant bioturbation of quartz sand grains. Here we have examined in detail the potential effects of one episode of ant nest-building on the age overestimation of affected sediments. Extrapolating the experimental results demonstrates that in sandy sites older than 5000 years, singlegrain small single-aliquot should be employed to methodology identify potential effects associated with bioturbation-induced age overestimation.



Pogonomymrex badius ants emerging from their central tunnel onto a chamber floor previously contaminated with colored grains.

# AMS Radiocarbon and Single-Grain OSL dating of Prehistoric Canals, Tucson Arizona: An example of when OSL works better than Radiocarbon dating

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Fertile river valleys in the southwestern US served as focal points for northward expansion of early agricultural activities. Key questions in SW archaeology research are related to when and how people transitioned from a foraging to an agricultural lifestyle following the arrival of maize from Mexico ~2100 BC. Evidence for the development of labor-intensive farming infrastructure such as canal systems can provide clues to the timing of this transition to larger population centers and dependence on cultivars for food supplies. Single-grain OSL and AMS radiocarbon results from a study of three prehistoric canals from the Santa Cruz River valley in Tucson Arizona are presented.

AMS radiocarbon samples (n=12) were collected from charcoal fragments from within the canal stratigraphy and floodplain strata cross-cut by the canals. Initial AMS results were quite exciting and suggested that these were the oldest canals within North America (~3500 <sup>14</sup>C yr BP, or ~1900-1500 BC). Later AMS results from taxonomically identified charcoal suggested younger ages (~3400-2600 <sup>14</sup>C yr BP, or ~1800-500 BC), but produce a wide range of possible ages.

Samples for single-grain quartz OSL dating (n=5) were collected from canal sediment to help improve age control. Target sediments were carefully chosen to avoid bioturbation and to select for sediment likely to have been exposed to light prior to deposition. Quartz sand was analyzed for single-grain dating using the single-aliquot regenerative (SAR) technique (Murray and Wintle, 2000) on at least 1000 grains and ages were calculated using a minimum age model. OSL results are internally consistent and indicate that one of the canals was in-use ~2.5 ka (~500 BC) and the other two canals were active ~2.3 ka (~300 BC).

OSL ages are consistent with AMS ages from individual charred seeds and plant stems from within the canal deposits (n=2) and subjacent floodplain sediments (n=3). However most AMS ages are inconsistent with OSL ages and regional archaeological context due to fluvial redeposition of charcoal, producing age overestimates of over 1000 years in cases. Moreover, AMS ages from the same canal, presumably in-use for less than a century, have age ranges spanning ~1300 years, while OSL central point age ranges are 200 and 700 years. Given the evidence for significant age overestimates from fluvially redeposited charcoal it is recommended that chronologies do not rely solely on radiocarbon ages from these settings. Moreover, this study has shown that while standard error (precision) is lower with AMS dating, single-grain OSL dating provides higher accuracy in this setting. It is recommended that both AMS radiocarbon and single-grain OSL dating be applied to similar canal settings in order to detect redeposition of older charcoal and to produce the most accurate chronology of canal use.

# Refining Iron Age chronologies in South Africa using luminescence: an archaeological case study from Melora Hill

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The area around Melora Hill in northeastern South Africa is rich with Late Iron Age (1300-1850 CE) archaeology and has been studied since the 1980s. *Melora Hilltop* is a large stone-walled site. The defensive style of walling and evidence from excavations associates the site with early 17<sup>th</sup> century Nguni farmers. *Melora Saddle* is at the southwestern foot of this prominent hill and is characterized by more than 50 hut floors exposed by sheet erosion, some of which were burnt. Only limited salvage excavation has been undertaken here, but evidence of a European glass trade bead from a perinatal pot burial indicated the settlement was from the mid-19<sup>th</sup> century.

In 2010, during a field trip to collect dosimeters and luminescence samples, a newly eroded whole *in situ* ceramic vessel was discovered in a Melora Saddle hut floor. According to its Eiland-type decoration, the vessel dates to the Middle Iron Age (1000-1300 CE). This discovery confounds thirty years of interpretation regarding a 250-year sequence of occupation at Melora Hill. Results from luminescence dating refine this chronology and offer researchers reason to re-examine the archaeology there.

Explaining Change in Production and Distribution Pattern of Olivine-Tempered Ceramics in the Arizona Strip and Adjacent Areas in the American Southwest using Optical Luminescence Dating

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The ceramic assemblage in the Arizona Strip and adjacent areas in Utah and Nevada is characterized by widely-distributed ceramics tempered with olivine, a volcanic mineral, between A.D. 100 and 1300. The source of this olivine is thought to be at Mt. Trumbull and Tuweep, near the northwest rim of the Grand Canyon. Olivine-tempered ceramics are distributed westward from these olivine source areas over a range of more than 100 km. The ultimate goal for this study is to understand the evolution of production and consumption patterns of olivine-tempered ceramics among a small-scale society within unstable agricultural environments. To investigate the source of olivine-tempered ceramics, chemical analyses on ceramic samples and clay including INAA and Laser ICP-MS (LA-ICP-MS) were used. Eight compositional groups were found in the 1069 ceramic data from Mt. Trumbull and the lowland Virgin area, which is the one of the destination of olivine-tempered ceramic trading. In this study, I would examine how the use of each clay group changed over time using optimal luminescence dating on the sherds with compositional data.

Application of OSL for the non-destructive dating of Native Remains

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The intact skeletal remains of a Native American were discovered in 1973 when they began to erode from the banks of Cimarroncito Creek located on the UU Bar Ranch, Colfax County, New Mexico. A young archaeologist from the adjoining Philmont Scout Ranch hastily recovered the remains and curated them in the Philmont Museum, where they subsequently became lost amid the collections for almost 36 years. In 2009, the newly appointed curator of the museum re-discovered the remains and asked faculty at New Mexico Highlands University to aid in the repatriation of the skeleton to the appropriate Native American tribe. Because no artifacts were recovered with the remains, it was almost impossible to fit the skeleton into a historical regional cultural chronology, which in turn made it difficult to determine the proper tribe to consult regarding repatriation under the Native American Graves Protection and Repatriation Act, or NAGPRA (25 U.S.C. 3001 et seq.). It seemed that the best options were to try to date either the deposits the skeleton was found in (if the original site could be located) or determine if the skeleton itself could be dated using non-destructive techniques.

Upon examination of the cranium, it was determined that the skull cavity had become filled with sediments during burial. These sediments could be easily sampled with a drill by accessing natural holes within the skull and no part of the skeleton would be damaged. The sediment was hard-packed into the skull, providing a degree of confidence that even though it would have been light-exposed while being recovered from the burial site, only the top portions of the sediment would have to be removed to get to a light-tight core. Two 20 g samples were obtained from coring by placing the skull upside down and, via the foramen magnum, gaining access to the tightly packed sediments inside. A hand drill was used to bore two ca. 7-8 mm holes to a depth of ca.10 cm. The sediments from the bottom ca. 5 cm of the boreholes were obtained without damaging or touching the skull.

The source of the sediments was the Sangre de Cristo Formation "red beds" that are composed predominantly of sandstone and shales, so even though the recovered cores were very small, they contained a fair amount of quartz. The sediments in the skull had a deep red color due to iron staining present on the grains. Moreover, the skull itself was stained red by exposure to the mineral-laden sediments and water and we were able to determine that the sediments were fluvially derived and not alluvial. Using Optically Stimulated Luminescence (OSL) dating methods, we were able to determine the minimum age of the sediment filling the skull, which will prove invaluable in repatriation efforts, as it provides a close approximate time of burial. The dose rate was determined by Inductively-Coupled Plasma Mass Spectrometry (ICP-MS) and the equivalent dose by Blue-light OSL on fine sand-size quartz using SAR protocols. The age of the skull was determined to be no older than 855 A.D. and no younger than 1455 A.D. The errors on the measurement are 5 to 6 percent. The specific advantage in burial contexts such as this is that OSL provides researchers with a nondestructive method for dating remains and for placing them in their proper cultural context, a particularly important matter in the case of Native remains. Given the success of this project, we suggest that under certain situations it may be advisable to routinely collect and preserve sediment samples trapped within skeletal remains or sediment adjacent to the burial.

What equivalent dose distributions and luminescence sensitivity are telling us about the sedimentary dynamics and sediment sources of the Amazon rivers?

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The Amazon fluvial system is the largest drainage network (7,050,000 km<sup>2</sup>) of the Earth, draining areas with different bedrock, tectonic regime, relief, climate, vegetation and land use and showing high variability in the type, yield and flux of sediments. The lower portions of the Negro, Solimões, Madeira, Tapajós and Xingu rivers stand out due to their distinct fluvial morphology and dynamics. Here, we investigate the relation between equivalent dose distributions and sedimentary dynamics of the Solimões, Negro and Amazonas rivers. Equivalent doses were determined in quartz aliquots using a SAR protocol. The sands of the Negro and Solimões rivers were also compared through their optically stimulated luminescence (OSL) sensitivity. The sands from the Negro River present equivalent dose distributions with a single mode, suggesting well-bleached sediments. The Solimões and Amazonas rivers present sands whose quartz aliquots show good performance in dose recovery tests but give dose distributions with multiple modes, indicating poorly bleached sands. Aliquots of the same sample of the Solimões and Amazonas rivers can present equivalent doses varying by three to ten times. Exposition of quartz aliquots to sunlight indicates that the sands from all rivers can be easily bleached. Thus, the different dose distributions observed in the Negro, Solimões and Amazonas rivers is attributed to differences in fluvial dynamics and not to the presence of hard-to-bleach quartz crystals. The Solimões and Amazonas rivers are characterized by high suspension load with turbid waters and sand transport in the central portion of the channel where water depth can reach up to 110 m during the wet season. Equivalent dose distributions with multiple modes would indicate mixing of sediments with distinct temporary storage in deep zones of the channel or in old bars since their last event of solar exposure in shallow water zones. The dating of deposition events in the Solimões and Amazonas Rivers requires the application of age models. The single-mode dose distributions presented by the Negro River sands are distinguished from common poorly bleached fluvial sands, being in agreement with their particular way of transport and deposition in channel margins due to wave reworking as indicated by geomorphological features and sedimentary structures. Preliminary data show that the sands from the Negro River present OSL sensitivity relatively higher (mean=18.4; stand.dev.=1.3 counts/sGy) than the sensitivity of the Solimões sands (mean=13.4; stand.dev.=2.7 counts/sGy). Despite the longer length of the Solimões River favors the increase in sensitivity due to a higher number of irradiation-bleaching cycles, the higher sensitivity of the Negro sands is attributed to their higher initial sensitivity inherited from nearby Cretaceous sandstones, which dominate the sediment input in the lower reach of the Negro River, as well as the wave action and clear water, which favors the sand transport through shallow zones able to bleaching even in the flooding wet season. The lower average and high variability of sensitivity of the Solimões sands would be related to Andean source areas dominated by diverse metamorphic and igneous rocks and difficult bleaching due to the sand transport under turbid water in the central deeper zones of the channel. Equivalent dose distributions and OSL sensitivity combined can be useful to understand how fluvial systems capture, transport and storage sediments.

Methods to reduce sample carrier contamination for luminescence measurements

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# **ABSTRACT**

Equivalent doses derived from sample measurements void of signal contamination are essential for accurate optically stimulated luminescence (OSL) measurements. We identified dose-dependent luminescence signals originating from stainless steel cups used for OSL measurements. Both new and previously used stainless steel cups produced signal above background levels. The luminescence signal most likely arises from a chemical film derived from a reaction that takes place upon heating and irradiation between the sample, silicone oil spray, and the cup itself. The film appears to accumulate between measurements due to its impervious nature to standard cleaning methods. While signals from sample carriers are typically small sources of error they may strongly contaminate the signal from low sensitivity samples. We tested several cleaning methods for both used and new stainless steel cups by measuring the OSL signal after increasing irradiation doses. The cleaning methods that produced the least amount of signal from the cups were combined to create two modified cleaning procedures that are effective in minimizing the unwanted luminescence signal. These cleaning methods incorporate the use of HF. Alconox (a detergent for silicone oil removal), and methanol. The cleaning methods are capable of reducing the luminescence signal of empty stainless steel cups to near background levels. Comparison of samples dated using sample carriers prior to and after the adoption of the modified cleaning procedures suggests significant equivalent dose underestimation with the use of sample carriers with dosedependent signals.

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What can feldspar TL tell us about thermal history?

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Understanding the thermal history of natural and anthropogenic materials such as sediments, rocks, ceramics and aggregates has important applications in many areas of the geosciences, in archaeological contexts, and in remediation assessment of modern fire-damaged structures. Such thermal history events are recorded in feldspar TL signals. In terms of TL process, the effect of thermal exposure can be simulated with simple first-order kinetics of isothermal decay or phosphorescence, and is well-matched by empirical observations from isothermal annealing experiments. Feldspar TL glow-curves have been interpreted to be a composite of many thermodynamic components from continuously distributed trapping sites, enabling their use from ambient temperature environmental/climatic contexts to high temperature thermal exposure involving several hundred degrees. The initial rise of the lowest glow-temperature peak following irradiation and thermal treatment can supply a quantitative indicator of thermal exposure, which relates to a combination of temperature and duration variables. In naturally accumulated signals thermal history is assessed by combining information from glow-peak position, dose comparison from plateau tests, and calibration measurements.

The thermometric properties of feldspar TL will be discussed and illustrated with applications such as Neolithic ceramic firing interpretation, evaluation of stratified luminescence dates, and monitoring of modern fire-damaged structures.

The Good, the Bad, and the Ugly: Single-grain dating of Kanab Creek Arroyo Sediments in Southern Utah

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Kanab Creek originates on the Paunsaugunt Plateau in southern Utah, flows through the Grand Staircase region of the Colorado Plateau and becomes a tributary to the Colorado River in the Grand Canyon, northern Arizona. Our research focused on a stretch of river where Kanab Creek flows through a canyon of Jurassic Navajo Sandstone and exists into a broad basin afforded by the Jurassic/Triassic Kayenta formation near Kanab, UT. In this reach, Kanab Creek transitions from a bedrock channel with alluvial terraces to an impressive 30-40 meter deep arroyo (incised channel) entrenched into thick fine-grained valley-fill sediments. Near-vertical and laterally continuous arroyo walls provide ample opportunities to sample fine-grained quartz-rich sediment for optical dating. Research objectives were to use OSL, combined with radiocarbon dating, to provide age control on the style and timing of past floodplain aggradation and arroyo entrenchment events in Kanab Creek.

The quartz sampled for optical dating of the Holocene arroyo sediments are derived from mature, quartz-rich bedrock. Due to its location on the south-central Colorado Plateau, Kanab Creek is an ephemeral fluvial system that experiences large discharge fluctuations due to seasonal monsoonal and rapid snowmelt runoff. In addition to the modern climate regime, the sedimentology and stratigraphy of terrace and basin-fill sediments suggests high energy deposition over the middle to late Holocene. Due to the high sediment supply, short transport distances and semiarid climate with flashy discharge, partial bleaching (zeroing) of the luminescence signal was expected to be a problem for OSL dating in this rapidly aggraded system. Therefore, single-grain dating and/or the use of a minimum age model were used in order to isolate the small population of grains that were zeroed prior to deposition. Methods for assessing the use of a minimum age model include overdispersion (>25%) and strong positive skew of the equivalent dose (De) distribution, comparison to radiocarbon ages derived from charcoal, and depositional facies. We found that sedimentary facies and bed thickness are important factors for minimizing the effects of partial bleaching. Thin, decimeter-scale plane bedded and ripple cross-bedded sandy lithofacies were found to be the best target for OSL dating. Additionally, results generally indicate that better bleached sediments are found in downstream reaches. Over the middle to late Holocene, Kanab Creek has incised and aggraded at least four times creating alluvial fill packages Qa4 (6.7-3.5ka), Qa3 (~2-~1.3ka), Qa2 (0.9-~0.3ka), and Qa1 (post-1880's incision). These fills are important for comparisons with regional chronostratigraphy and paleoclimate records to better understand these unique fluvial systems.

OSL dating of fluvial terraces in NW China: comparison of grain sizes

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We use optically stimulated luminescence dating to determine ages for faulted and folded fluvial terraces in the western Tarim Basin in NW China. Previously dated fine-grained samples from terraces in the area were interpreted to have had significant partial bleaching, resulting in ages that we suspected were not the true depositional ages. To more accurately date the terraces, we compared ages from two different quartz grain sizes, 4-11 µm and 90-180 µm, at sites on two fluvial terraces. At each site, we collected four OSL samples at different depths, in addition to collecting a <sup>10</sup>Be depth profile nearby on the same surface. Fine-grained and coarse-grained samples were measured using the SMAR and 2-mm smallaliquot SAR protocols, respectively. Coarse-grained samples had equivalent doses with highly skewed populations, and we used the minimum age model to calculate ages. Our results from younger (<50 ka) fluvial terraces suggest that coarse-grained samples more accurately reflect the depositional age of the terrace, whereas fine-grained samples from the same site may overestimate the age by as much as ~100%. For example, fine-grained ages for one terrace are 24-39 ka, whereas coarse-grained ages are 14-18 ka. We suggest this discrepancy arises from: (1) the low sensitivity of some quartz grains, due to the short transport times in the fluvial system after erosion from bedrock in the surrounding growing orogens; and (2) the fine-grained fraction is transported quickly through the fluvial system predominantly as suspended sediment. The sediment-laden waters have high turbidity, and as a result the suspended sediment is not exposed to light long enough to be fully bleached. In the western Tarim Basin, suspended sediment in the rivers is often a result of seasonal floods and erosion of the older geomorphic surfaces and weakly-consolidated, exhumed Tertiary bedrock. The coarse-grained fraction is more likely to be transported as channel sands, to be temporarily stored in sandbars, and to take longer to travel through the fluvial system, and, therefore, is more likely to be bleached. We note that the highly-skewed equivalent dose population of the coarse-grain sediments suggests this fraction is not fully bleached either. Selecting the appropriate grain size is important because variations in depositional ages calculated from different grain sizes have implications for the chronology of geomorphic surfaces and derivative products, such slip rates that are based on them. Based on the results of our study, we suggest using small-aliquot coarse-grained dating for fluvial terraces in tectonically active orogens.

# Impact of feldspar contamination for OSL dating on aeolian and fluvial sand in Illinois

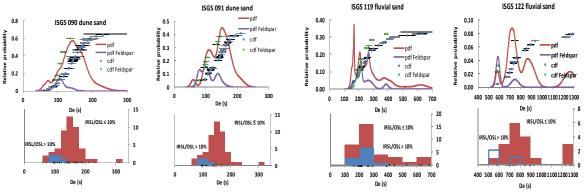
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Quartz and feldspar grains from the same sample may yield different equivalent dose (De) because feldspars receive or lose charges from their internal dose or abnormally fading. Quartz grains, if contaminated by feldspar when single aliquot regenerative (SAR) protocol is used, can yield over or underestimated De values thus OSL dates depending upon the geographic locations. We investigated the influence of feldspar contaminates on dune and glaciofluvial sand from the Illinois River valley. For extracting and purifying quartz: samples were wet sieved to extract 150-250 µm fractions; treated with 2M HCl and bleach overnight for each treatment; quartz grains were separated using a density of 2.70 g ml<sup>-3</sup> of lithium metatungstate liquid and etched using 48% HF followed by 2M HCl for 50-110 minutes for each treatment. Yet, we occasionally found that aliquots contained significant amounts of feldspar, indicated by their IRSL/OSL ratio.

Using the central age model, we calculated and compared De values of quartz with and without feldspar contaminate, defined as IRSL/OSL to be greater than 10%. The dune sands selected here for comparison yielded De values ranging from 17 to 20 Gy and glaciofluvial sands yielded De values from 32 to 130 Gy. It appears that feldspar contaminated aliquots reduced the De values in all occasions (Table 1, Fig. 1). Results suggested that when measuring quartz OSL signals using the central age model, feldspar contamination could underestimate OSL dates for either dune or glaciofluvial sands in the Illinois River valley. It appears that abnormal fading rather than the internal dose causes the reduction in De values.

Table 1. De (Gy) values obtained from dune and fluvial sand in Illinois Valley

Lab#	# aliq.	IRSL/OSL ≤ 10%	IRSL/OSL >10%	# Cont. Aliq.	Dose rate	Materials
ISGS 090	71	20.57	15.48	5	1.64	Dune sand
ISGS 091	47	19.92	14.99	9	1.53	Dune sand
ISGS 095	60	19.58	10.41	20	1.20	Dune sand
ISGS 096	60	19.26	12.28	24	1.15	Dune sand
ISGS 100	83	17.54	12.33	44	1.12	Dune sand
ISGS 119	48	38.11	32.26	11	0.98	Fluvial sand
ISGS 122	29	116.01	88.55	3	0.80	Fluvial sand
ISGS 123	41	130.51	123.96	17	1.08	Fluvial sand
ISGS 124	41	132.82	128.27	10	1.20	Fluvial sand



**Fig. 1.** De (seconds) of quartz aliquot with (purple) and without (red) feldspar from eolian and fluvial sands in Illinois River valley. pdf - probability density function; cdf - cumulative density function.

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# Success in OSL dating of proximal glacial sediments from the South Island, New Zealand and Olympic Mountains, Washington

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This study presents optically stimulated luminescence (OSL) ages from glacial deposits on the South Island of New Zealand and the Olympic Mountains of Washington. OSL dating of glaciofluvial and glaciolacustrine sediments has been met with variable success in past studies. The greatest issue with dating glacial deposits has been incomplete bleaching due to subaqueous and sub-ice sediment transport and high sediment loads typical of glacial environments (Fuchs and Owen, 1998). Another primary complication relates to poor luminescence properties of quartz because the sediment has been eroded directly from bedrock and has not been sensitized (e.g. Preusser et al., 2006; Pietsch et al., 2008; Alexanderson and Murray, 2009). Ice-proximal settings should exacerbate both of these problems. Additional complications include thermal transfer (e.g. Rhodes, 2000; Alexanderson, 2007; Klasen, 2007) and feldspar contamination (e.g. Lukas et al., 2007). Moreover, previous work has highlighted problems with OSL dating of quartz from the South Island of New Zealand. Issues addressed include poor luminescence properties and evidence of thermal transfer attributed to short transport distances, which may not be fully corrected for in laboratory protocols (Preusser et al., 2006).

This study analyzes OSL samples from exposures of glacial sediment in the South Fork Hoh River valley of the Olympic Mountains, Washington and the Lake Hawea valley, New Zealand. In the Olympic Mountains, Pleistocene radial valley glaciers flowed from the high peaks to the coastal lowlands (Thackray, 2008). Today, the remnants of these glaciers and their meltwater feed the Hoh and South Fork Hoh rivers, which flow northwest out of the mountains. Cut banks along the South Fork expose numerous outcrops of glacial sediment. Glacial cycles represented in the stratigraphy of these exposures include the Hoh Oxbow II (>23 ka), Twin Creeks I (~19 ka), and Twin Creeks II (<17 ka) advances. Lake Hawea lies on the eastern side of the Southern Alps drainage divide in northwestern Otago and occupies a north-south trending glacially excavated overdeepened trough. Glacial deposits of the >16 ka Hawea advance (McKellar, 1960) at the southern margin of the basin impound the modern lake and are exposed in shoreline bluffs.

Samples for OSL dating were collected from sand lenses in proximal glacial outwash sequences and glaciolacustrine deposits at both locations. Standard laboratory procedures were followed with the single-aliquot regenerative (SAR) protocol on quartz grains. Although some samples show mild partial bleaching and equivalent dose overdispersion, these problems will be mended by decreasing aliquot size and use of a minimum age model.

Despite potential problems, preliminary results from three samples from the South Fork Hoh River valley, WA and two from the Lake Hawea valley, NZ are in stratigraphic order and agree with radiocarbon ages. Samples generally show good signal decay curves, little to no feldspar contamination, and good quartz sensitivity.